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Physical, Mechanical properties and Internal Structure Characteristics of Cement-Locust Bean Pod Waste Ash Mortars

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ABSTRACT

The need to reduce the environmental pollution resulting from agro-wastes and to source a material that can be used to replace cement in order to reduce the pressure on its consumption necessitated this research. This study was designed to investigate the effect of cement replacement with locust bean pod ash (LBPA) as a supplementary cementitious material on compressive strength of mortars. The fresh properties (workability, initial and final setting times), compressive strength at 7, 14, 21 and 28 days and microstructural analysis Scanning Electron Microscope (SEM), Energy Dispersive Spectroscopy (EDS), and X ray diffraction analysis (XRD) were evaluated. LBPA were considered at replacement levels of 0, 10, 15, 20 and 30% of cement mass for preparation of the mortar samples. Workability of the cement mortars reduced as the content of LBPA increases while initial and final setting times increased in relation to increase in LBPA content in the matrix. A decrease of about 98% in compressive strength occurred at 30% LBPA and after 14 days of curing and the highest increase in compressive strength was recorded at 15% LBPA after 28 days of curing. Maximum compressive strength ranged 38.3 and 65 MPa after 7 to 28 days curing. Microstructural analysis revealed less voids and pores, and the presence of dense Calcium-Silicate-Hydrate (CSH) gels which helped to maintain the optimum compressive strength at 15% LBPA cement replacement level of the mortar. The results indicated that LBPA is a reactive pozzolanic material and can be used as a supplementary cementitious material for producing medium-strength concrete.

Keywords: Compressive; crystalline; agro-waste; network; ash

Introduction

A pozzolan is described as a siliceous or aluminosiliceous (aluminous and siliceous) material which in itself, possesses little or no cementitious value but which will, in finely divided form and in the presence of water, react chemically with alkali and alkaline earth hydroxide at ordinary temperatures to form or assist in forming compounds possessing cementitious properties (Omoniyi and Akinyemi,

2012). Pozzolanic materials will form calcium silicate cement when they react with soil particles in the water. The cementing agents are the same as in the case of Portland cement, however, in Portland cement, the calcium silicate gel is formed from the hydration of anhydrous calcium silicate (cement).

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Due to increasing industrial and agricultural activities, tonnes of waste materials are deposited into the environment with little effective method of waste management or recycling. Some of these deposits are not easily decomposed and their accumulation is a threat to the environment and people at large. Some of these waste materials are locust bean pods, bagasse, rice husks, sugarcane straw, bamboo leaf, maize cobs, snail shells, palm-kennel shell, coconut shell, saw dust and groundnut shell (Omoniyi and Akinyemi, 2012; Adama and Jimoh, 2012).

Super plasticizers are chemical admixtures used where well-dispersed particle suspension is required. These polymers are used as dispersants to avoid particle segregation and improve the flow characteristics of suspensions in concrete applications. Their addition to concrete or mortar allows the reduction of the water-cement ratio, not affecting the workability of the mixture, and enables the production of self-consolidating and high-performance concrete.

Population growth has led to an astronomical increase in demand for cement as it has indirectly lead to increases in the construction industries I terms of housing and other social facilities. The increase in construction has increased the demand for cement, sand, gravel and other construction. Above all, the demand for cement is more than other construction materials. Therefore, in order to meet this demand, cement productions have been jerked up with its accompanied carbon emissions (Adama and Jimoh, 2012; Omoniyi and Akinyemi, 2012). Supplementary cementitious materials (SCM) have been proffered as the solution to this problem through its effective and successful use in construction materials. The adoption of SCMs in construction leads to a major reduction in carbon emissions into the environment (Akpenpuun *et al.*, 2017; Mahmud *et al.*, 2016; R blier *et al.*, 2013). Nonetheless, the accessibility of these substances are limited to specific locations and industrialization, therefore

it is imperative that focus must be shifted to conducting studies on SCM's emanating from agriculture

Locust bean tree (*Parkia biglobosa*) is a deciduous tree which grows in Sub Saharan Africa with an annual production of about 201,000 tonnes of the bean fruit in Nigeria annually (Sina and Traor , 2002). The pod is usually opened up in order to remove the pulp and the seeds encased within it. Thereafter it is discarded, burnt and the ashes dumped in landfills (Adama and Jimoh, 2012; Tangchirapat *et al.*, 2009). Limited studies had been conducted on its use as supplementary cementitious materials in concrete, however, majority of those studies paid no attention to microstructural analysis such as Scanning Electron Microscope (SEM), Energy Dispersive Spectroscopy (EDS), and X ray diffraction analysis (XRD) and non-use of superplasticizers which would have improved the workability of the mortars produced (R blier *et al.*, 2013). In this present experiment, the performance of locust bean pod ash blended cement on workability; setting time and compressive strength of mortars were evaluated while incorporating superplasticizer. Similarly, the use of SEM, EDS and XRD were employed to further analyse the internal networks and the crystalline materials located within the samples.

Materials and methods

Locust bean pod ash (LBPA)

The waste was obtained from a farm around the University of Ilorin permanent campus. Sorting was done by removing unwanted organic materials such as leaves, shrubs and stalks. LBP was sun dried for 14 weeks to reduce the moisture content. 10 kg of LBP was calcined in a muffle furnace at 600 C with heating rate of 40 C/min for 2 hours and 2 hours' retention time. The obtained ashes were ground manually for 4 minutes and the particle size analysis was done according to ASTM C 618 standard (Figure 1). The ash was

classified as Class C pozzolanic material. Shown in plates 1 and 2 are the pods and ash.



Plate 1: Locust bean pods

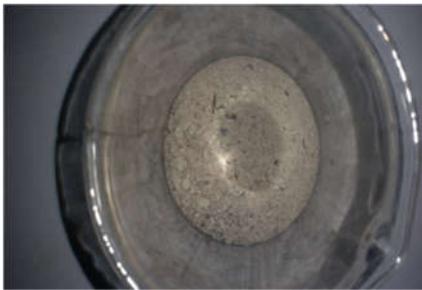


Plate 2: Locust Beans Pod Ash (LBPA) in beaker

Fine sand aggregate

Cement

Ordinary Portland cement that conforms to type 1 (ASTM C150 / C150M-18, 2018) was used and mixed with water, sand, LBPA and superplasticizer.

Admixture

The super-plasticizer (SP) used was Hydroplast – 300, a third generation high performing polycarboxylate ether super-plasticizer with great water reducing properties which allows for the production of high consistency mixed concrete with low water-cement ratio. It also ensures extended workability and improves early and final strength development (Akeem and Blessing, 2018).

Mixture proportioning

Replacement levels of 0%, 10%, 15%, 20% and 30% of cement mass were used. The water: cement binder ratio was maintained at 0.55 and a constant dosage of 1% of cement of SP was utilised in order to enhance the workability of the mortar. Mortar

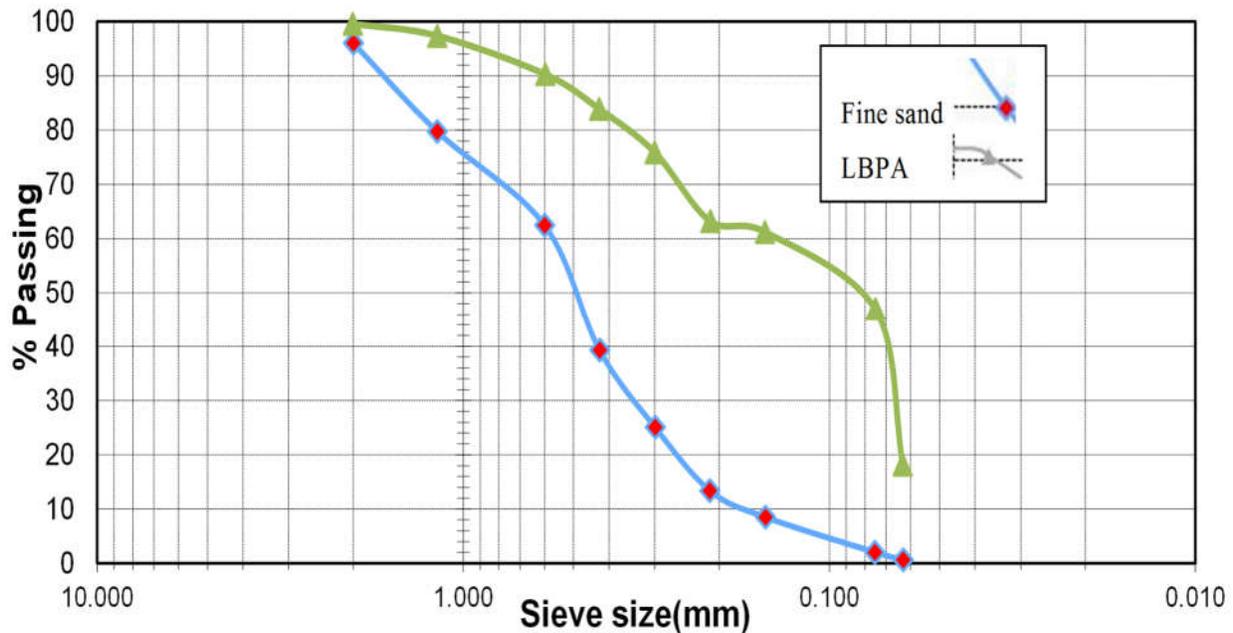


Figure 1: Sieve analysis of LBPA and sand

samples were prepared for scanning electron microscopy and energy dispersive spectroscopy (SEM/EDS) as well as for X-ray Diffraction (XRD) analysis.

Page | 3340 **Preparation and casting of samples**

Cement, sand and LBPA were mixed thoroughly until homogeneity was attained. The required quantity of super-plasticizer was dissolved in a little amount of water and then added to the cement-sand-LBPA mix. The mix was turned with hand-trowel until uniformity in colour and texture was obtained. Twelve samples measuring 50 by 50 mm were produced and cured for 7, 14, 21 and 28 days in a curing tank at 23°C respectively.

Testing programme

The adopted testing method used are setting time (ASTM Standard C807-13, 2013), workability (ASTM Standard C1437-15, 2015). In the same vein, compressive strength (ASTM Standard C109M-16, 2016) test was performed after 7, 14, 21 and 28 days of water curing.

Characterisation of blended cement mortars

The fractured samples of were examined using a field emission scanning electron microscope (FESEM) (model: JSM-7600F, Jeol, Japan) at an acceleration potential of 15 kV. The fractured surfaces of the specimens were sputter-coated with a thin layer of platinum using a JFC-1600 auto fine coater. The EDS results were obtained using the same model of SEM machine.

X-ray diffraction Analysis for Mineral Identification

Powdered samples were pelletized and sieved to 0.074mm. These were later taken in an aluminium alloy grid (35mm x 50mm) on a flat glass plate, covered with a paper and the samples were compacted by gently pressing them with the hand. Each sample was run through the Rigaku D/Max-IIIc X-ray diffractometer developed by the Rigaku Int. Corp. Tokyo, Japan and set to produce

diffractions at scanning rate of 20/min in the 2 to 500 at room temperature with a CuK α radiation set at 40kV and 20mA.

Results and discussions

Workability

The results of the fresh mortar properties of the LBPA mortars (Figure 2) showed that slump flow was in the range 14 to 49 mm which is within the range of 10 – 50 mm typically required for light reinforcement foundation. The least workability was gotten at the highest percentage of 30% LBPA, while the highest slump flow was obtained at the control mix (0% LBPA). This could be interpreted that workability reduces as the content of LBPA increases in the mortar mix. This reduction in flow was similarly reported by Tangchirapat *et al.* (2009), where it was stated that an increase in ash content led to a progressive decrease in workability. The slump values of samples A, B, D and E were 49, 40, 34, 29 and 14 respectively. This reduction in slump on increase of LBPA could be attributed to the porous nature which is due to the presence of macro and mesopores located within and on the surface of the material leading to its huge specific surface area. The LBPA will thereafter absorb some amount of water unto its surface during mixing which would cause subsequent reduction in free water and a lower slump value (Cordeiro *et al.*, 2012). The chemical composition (cement and LBPA) and the different mortar mixes are shown in Tables 1 and 2.

Table 1: Chemical composition of cement and LBPA

Oxides	LBPA, %	Cement, %
SiO ₂	40.25	20.70
Al ₂ O ₃	13.15	5.75
Fe ₂ O ₃	9.00	2.50
CaO	12.50	64.00
MgO	2.03	1.00
SO ₃	1.50	2.75
Loss of ignition	6.05	2.30
Blaine fineness (cm ² /g)	5502.62	3100.43

Table 2: Mix design of mortar (kg/m³)

Mixture code	Cement	LBPA	Sand	Water	SP(grams)
Control (C)	628.3		1256.4	341.5	
A	565.5	62.8	1132.3	314.2	5.7
B	534.1	94.2	1069.7	297.3	5.3
D	502.6	125.7	1006.1	279.2	5.0
E	439.8	188.5	880.2	244.3	4.4

A – 10%, B - 15%, C – 20%, D – 30%

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temperature during the ashing process, low temperature would yield crystalline powders such as from open burning while high temperature would produce amorphous powders (Tangchirapat *et al.*, 2009). The same trend was confirmed by other researchers that used rice husk and corn cub ash in concrete. Another reason adduced for this pattern is that the low rate of cement hydration in the paste consisting of the LBPA pozzolan led to an increase in both initial and final setting times on subsequent increase in cement replacement levels (Cordeiro *et al.*, 2012).

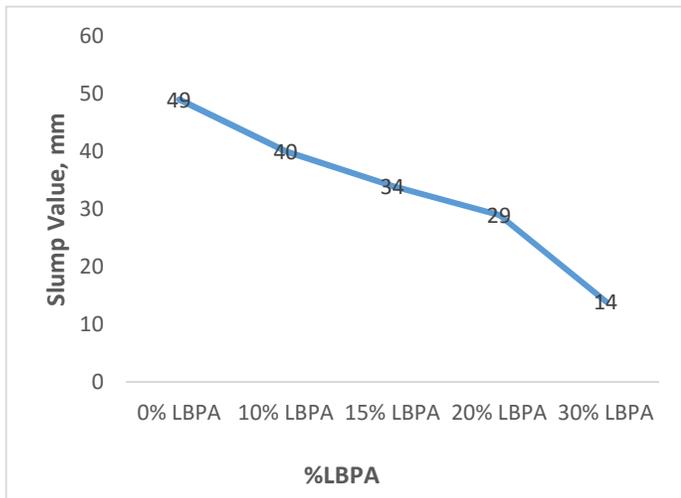


Figure 2: Slump values of fresh concrete

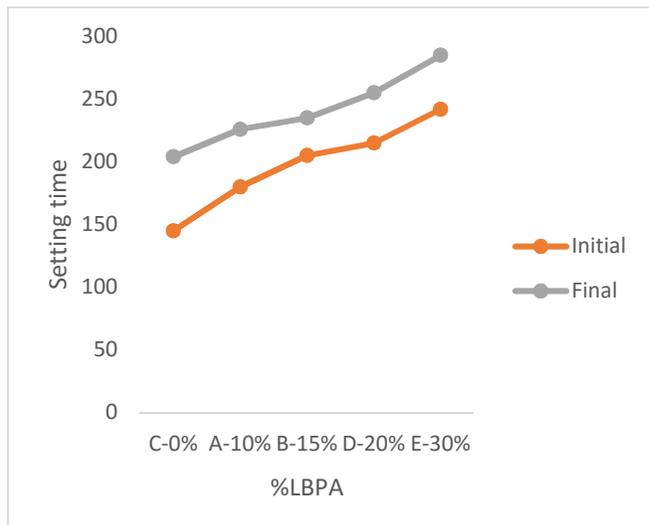


Figure 3: Initial and final setting times

Initial and final setting times

Figure 3 shows the final and initial setting times. The mean setting time was 174.5±41.7, 204±33.9, 220±28.3 and 263.5±30.4 for 0, 10, 15, 20 and 30% cement replacement with LBPA. An observed increase in both initial and final setting times as the cement binder replacement increases was noted. In relation to the control sample, 30% cement replacement with LBPA caused an increase of 66.8% in the initial setting time of the cement-LBPA-mortar, while an increase of 39.7% was observed in the final setting time. The fineness of the LBPA powder determines both setting times, this could either be amorphous or crystalline in nature. This is dependent on the

Compressive strength

The compressive strength in relation to the LBPA cement replacement levels and specimen age (7, 14, 21 and 28 days) is shown in Figure 4. The error bars indicate standard deviation from the average of three replicate samples tested. As seen in Fig. 4, there was an increase in strength up till the 15% replacement level then a reduction in the values was noticed. At 7 days the compressive strength ranged 21.3 – 38.4 MPa when cement replacement level was 0 to 15% however, from 20% to 30% cement replacement, a decrease in values from 24 to 23 MPa was observed. The results are comparable to Chao-Lung, Le Anh-Tuan, and Chun-Tsun (2011) who reported that

the optimum compressive strength was obtained at 15% cement replacement with rice husk ash, but decreased on increase in cement replacement beyond 20% . This increase in compressive strength is attributed to reduced pores and increased Calcium-Silicate-Hydrate (C-S-H) compound in the matrix which resulted into the formation of a dense mortars after the early curing days (Raman *et al.*, 2011). The data from this experiment was compared with similar studies conducted using agro-waste based ash (rice husk, bagasse) as cement replacement to relate the effects of cement replacement and the age to the compressive strength as shown in Table 3 (Omoniyi and Akinyemi, 2012; Raman *et al.*, 2011).

Table 3: Comparison with compressive strength of other agro-waste ashes

Agro waste ash	Optimal %	Super plasticizer	Age	Comp. Stre. (MPa)
LBPA- P.S.	15	Yes	28	65
Palm oil fuel	10	No	28	55
Rice husk	15	Yes	28	51
Bamboo leaf	20	No	28	56

P.S. – present study

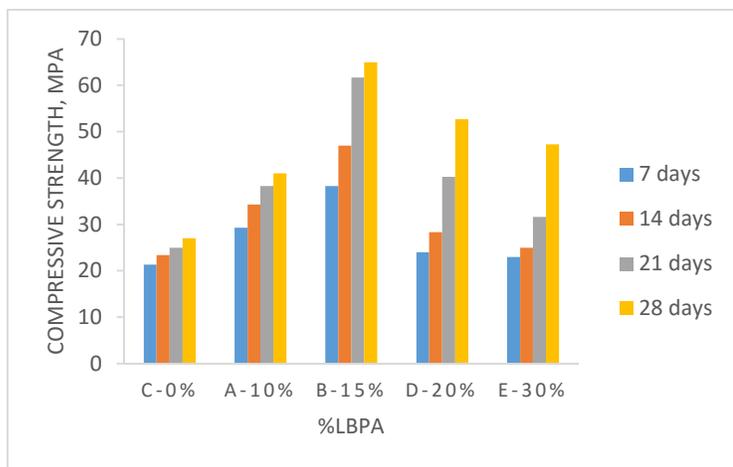


Figure 4: Compressive strength of LBPA blended mortars (C = control, A = 5%, B = 10%, D = 20%, E = 30%)

The basis for the determination of the compressive strength and strength activity index is their importance in the design of concrete and mortars for various load bearing applications. The mean compressive strength was 24.38 ± 2.58 , 35.75 ± 5.08 , 53.00 ± 12.54 , 36.35 ± 12.89 and 31.73 ± 11.01 for 0, 10, 15, 20 and 30% cement replacement with LBPA. It has been noted by some researchers that mortars incorporated with supplementary cementitious materials (SCM) tend to develop later strength which would be higher than the control samples without SCM replacement (Chopra and Siddique, 2015; Raman *et al.*, 2011).

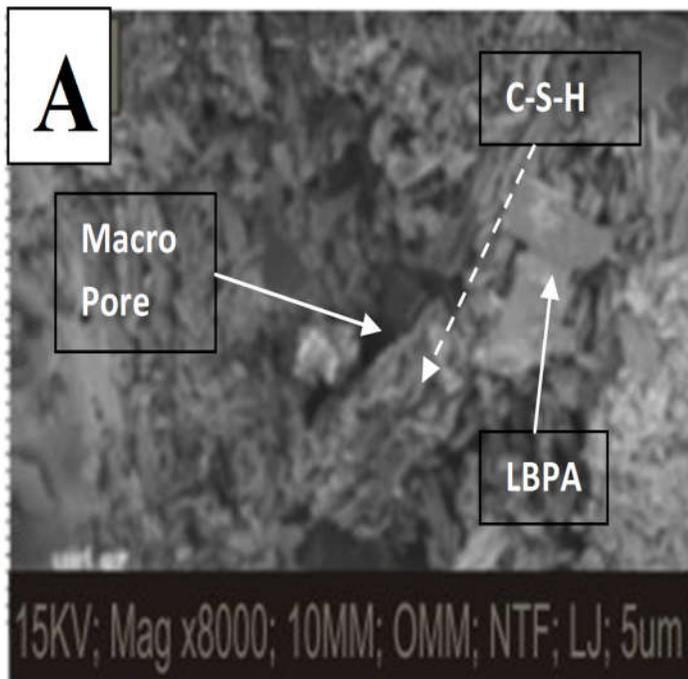
Characterisation of LBPA blended cement mortars

Scanning electron microscope (SEM)

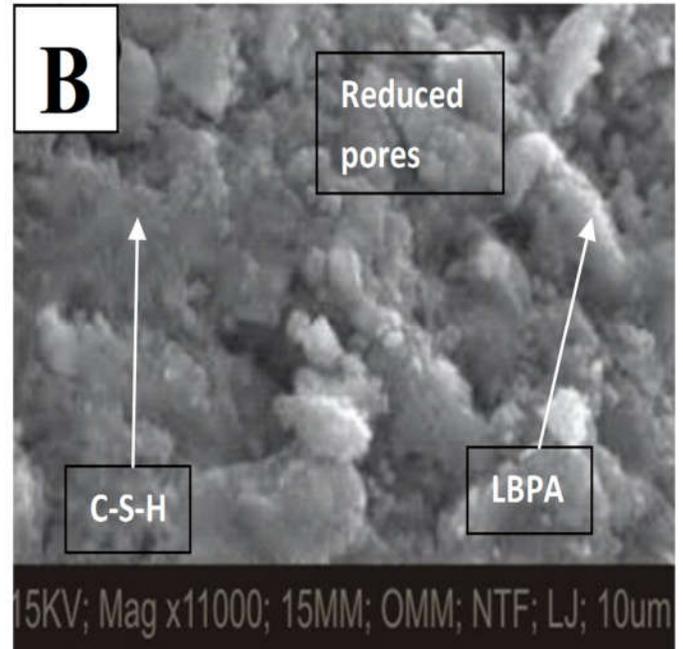
The micrograph of the surfaces of 10% (A), 15% (B) and 20% (D) LBPA mortar samples after 28 days of curing is shown in Figure 5. Fig. 5(a) showed presence of both micro and macro pores of sample with 10% LBPA content and C-S-H gel could also be sighted in it. These pores are largely caused by evaporation of water during hydration based on the LBPA content in the sample. It has been stated that agro based ashes used as cement replacement tend to absorb free water into its pores during mixing and this is released at the later stages when the relative humidity of the cement drops (Mahmud *et al.*, 2016). This should cater for the drop in water required for hydration to be completed but where this free water is not available in required quantity, porous surface may emerge as noticed in this result (Chopra and Siddique, 2015; Mahmud *et al.*, 2016). The presence of these voids largely led to the poor compressive strength performance of the sample. However, Fig. 5(b) shows that mix with 15% LBPA replacement had less voids on the surface and could be attributed to improved hydration, and likewise large occurrence of C-S-H gels could be noticed as well on the surface. This could be the reason for the optimal compressive strength

gotten at this mix level. The increase in LBPA content could possibly improve the pore characteristics because it led to higher amount of C-S-H gel formation during the pozzolanic reaction which contributed to the refinement of pore structure subsequently leading to volume reduction of large pores and porosity of the mortar. Fig. 5(c), is the micrograph of 20% LBPA replacement, it was noted the mix commenced disintegration at the quantity of LBPA was increased; this perhaps led to the reduction in compressive strength. Ettringites which are the needle like crystals that are developed in vacant spaces during hydration also led to weakening of the concrete strength. Some portions of incompletely hydrated cement particles the concrete strength. Some portions of incompletely hydrated cement particles were also identified, which also contributed to lower strength of the mortars.

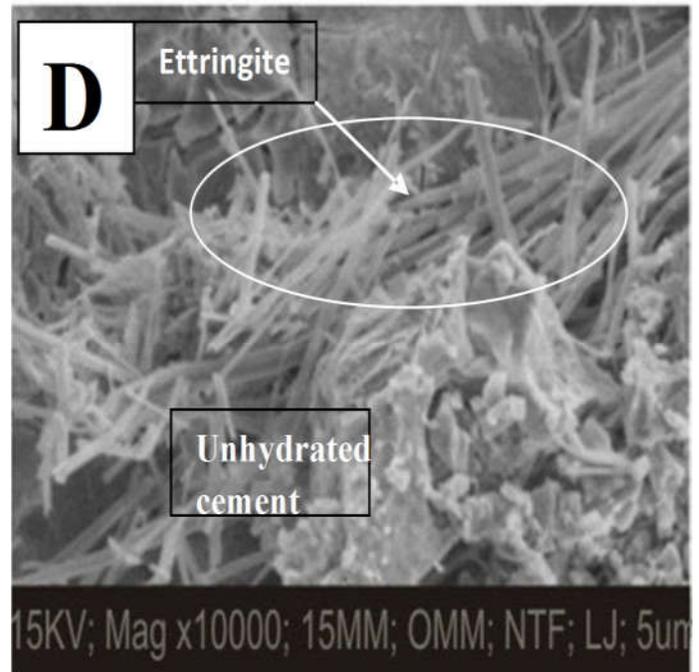
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(A) 10% LBPA
(15KV;Magx8000;10MM;OMM;NTF;LJ;5um)



(B) 15% LBPA
(15KV;Magx11000;15MM;OMM;NTF;LJ;10um)



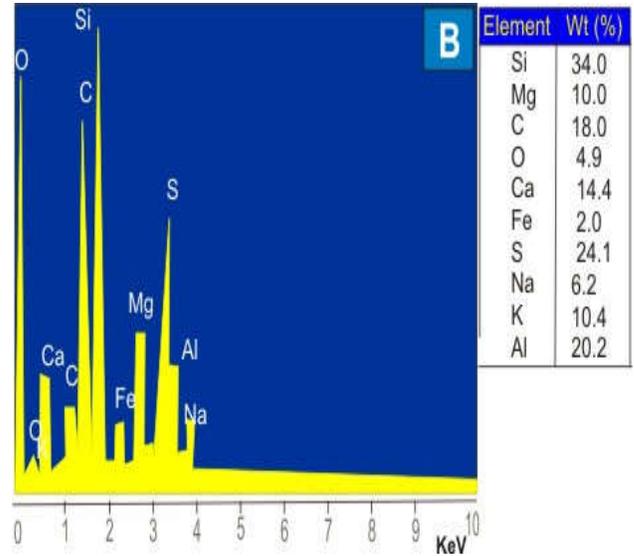
(C) 20% LBPA
(15KV;Magx10000;15MM;OMM;NTF;LJ;5um)

Figure 5: SEM images of mixes A, B and D

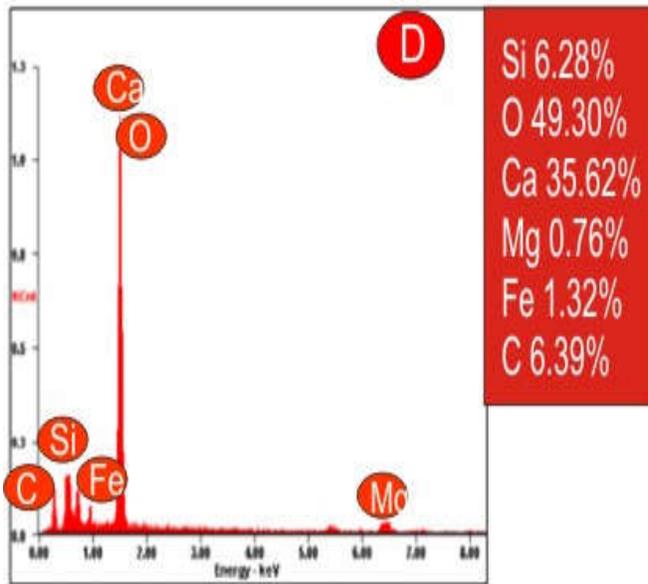
Energy Dispersive Spectroscopy (EDS)

It is seen in Figure 6 that mix B contains 34% of Silica whereas mixes A and D show 8.28% and 6.28% respectively. Also mix B contains 14.4% of calcium, while A and D are 35.60% and 35.62% as well. Only mix B had 20.2% of Alumina while the rest had none. This validates the presence of C-S-H in the SEM micrograph for mix B. The selected region in sample B had gel composition of Si/Ca = 2.36 and Al/Ca equivalent to 1.40. While only Si/Ca relationship of 0.23 and 0.18 could be noted for mixes A and D since Alumina was absent from their composition. This clearly shows that the presence of C-S-H gel compound in mix B greatly helped in improving the compressive strength much more than the other mixes. A clear difference in the proportion of Si/Ca gel phase among B, A and D could be clearly spotted.

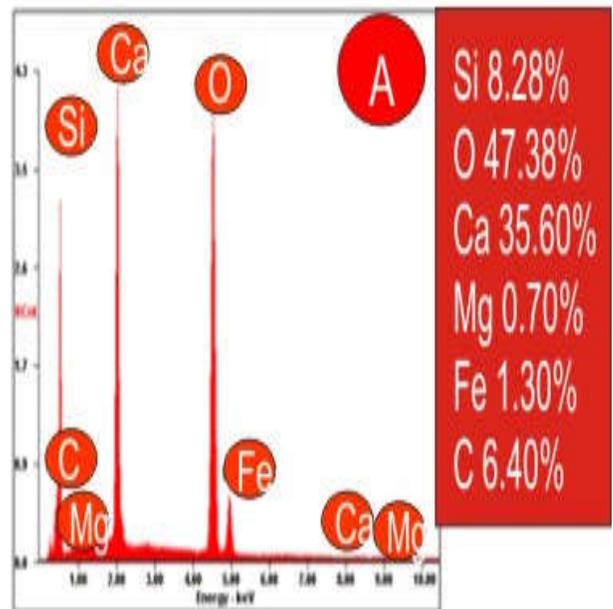
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(B) 15% LBPA



(C) 10% LBPA



(A) 20% LBPA

Figure 6: EDS of A, B and D mortar mixes

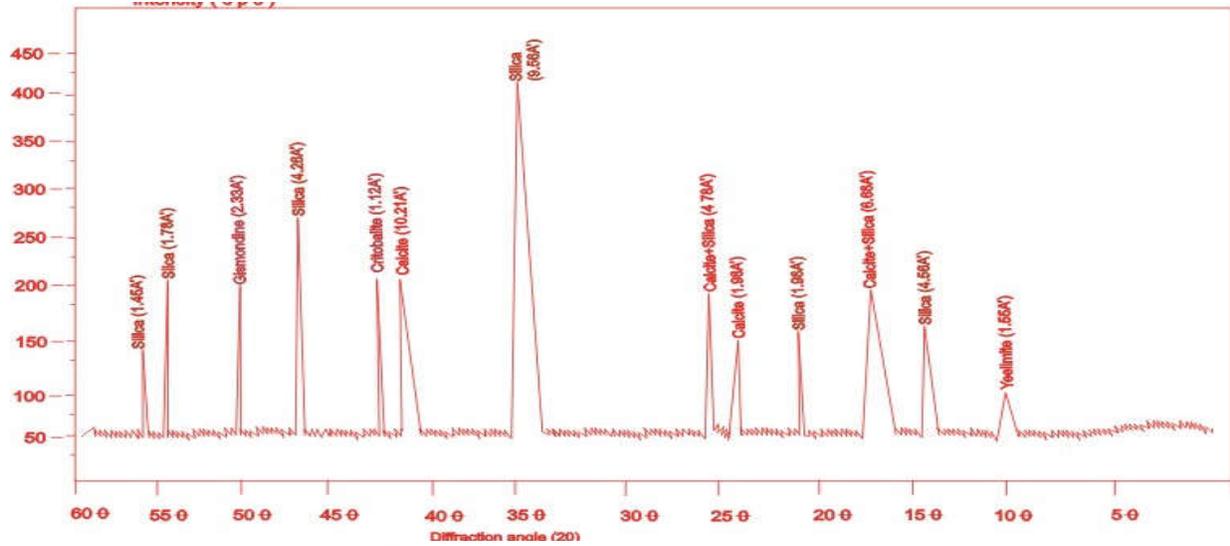


Figure 7: XRD analysis of sample A

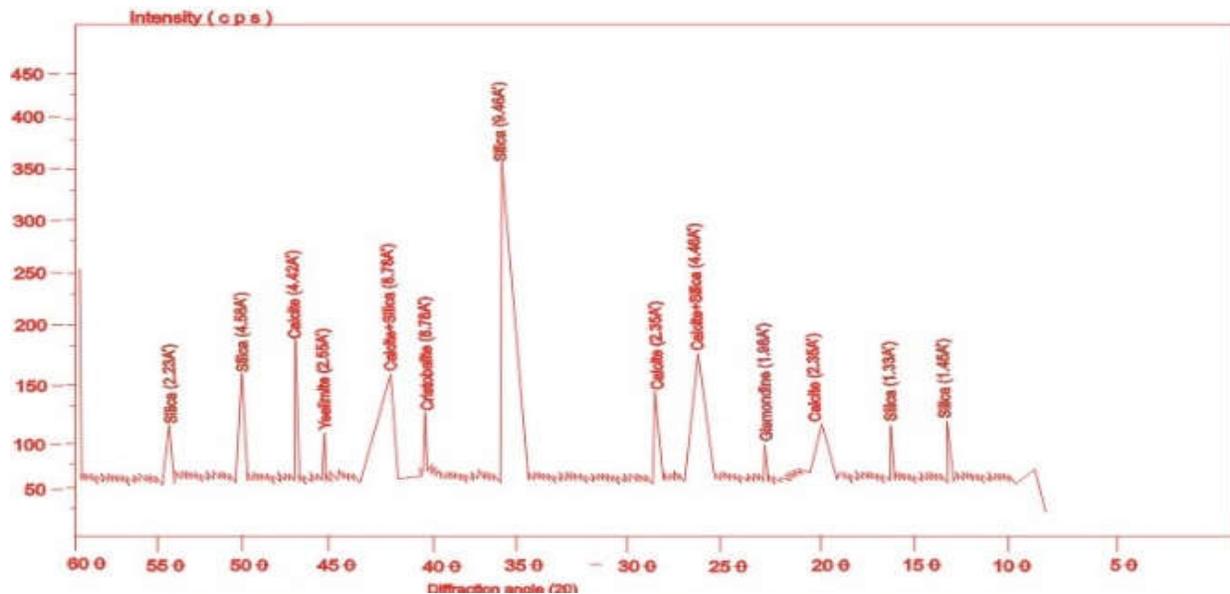


Figure 8: XRD analysis of sample D

X ray diffraction analysis

The XRD diffraction patterns of two samples A and D are shown in Figures 7 - 8. Sample A has calcite with silica which is the major peak at 17.5° and it is within the amorphous range between 10° and 20° respectively for the 2θ range. This is favourable for pozzolanic activity of the compound. Crystalline compounds such as silica, cristoballite and giesmondine are the other major peaks noted at 25.8° , 33° and 47° respectively.

However, sample D had major peaks at 27.5° , 35° and 42.5° which belong to crystalline compounds such as calcite and silica. These compounds played major role in the decrease of the compressive strength as seen from the compressive test because of their crystalline forms which originated from the slow rate of cooling of the LBPA within the furnace during the calcining process.

Conclusion

It can be concluded that, the workability of mortar reduced as LBPA level was increased in concrete mix. However, the setting time of the mix increased as the level of LBPA content increased.

Increase in LBPA content was observed to result to increment in compressive strength. The optimum cement replacement ratio was 15% and curing time of 28 days. The microstructural analysis showed the pronounced presence of CSH gel in the 15% LBPA replacement of cement and curing age of 28 days.

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